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VAPOR PRESSURE, LATENT HEAT OF VAPORIZATION, AND TRIPLE-POINT TEMPERATURE OF N_2O

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ABSTRACT

The vapor pressure of N_2O has been measured from the triple point ($T=182.351^\circ K=-90.809^\circ C$, $p=658.9$ mm Hg) to $236^\circ K$ ($p=10.25$ atm). The data are compared with previous work in a graph that includes the entire liquid range.

The latent heat of vaporization was measured from the triple point to $205^\circ K$. At the boiling point ($184.695^\circ K=-88.465^\circ C$) the latent heat is 16.55 kJ mole $^{-1}$. The consistency of the data was tested with Clapeyron's equation, which was also used to compute latent heats above the range of the direct measurements.

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I. INTRODUCTION

A survey of the literature on the vapor pressure of N_2O reveals the fact that the agreement between the results of different observers is poor. Much of the work is very old and shows differences of more than 20 percent, whereas among the more recent papers there are insufficient data to establish the vapor-pressure curve with any great degree of accuracy. The situation with regard to latent heats of vaporization is likewise unsatisfactory, as only a single value [1]¹ has been reported since 1900.

As the time that could be devoted to this investigation was limited, it was decided to dispense with the use of a piston gage, and to make all pressure measurements above 2 atmospheres with a Bourdon gage. This resulted in a considerable sacrifice of accuracy, but it is thought

¹ Figures in brackets indicate the literature references at the end of this paper.

that in spite of this the data are considerably more accurate than any previously available. Vapor pressures below 2 atmospheres were measured with a mercury manometer, while latent heats of vaporization were obtained with an adiabatic calorimeter.

II. MATERIAL

Commercial N_2O prepared for anaesthetic purposes was slowly admitted to a trap which was essentially an unsilvered Dewar flask. The inner tube of the trap was filled with liquid air. Solid N_2O collected on the outside of this tube, while noncondensable gases were pumped off with a diffusion pump. When about 25 g of N_2O had been collected the valve in the supply line was closed. After the vacuum became good, the line to the vacuum pump was also closed and the trap allowed to warm up. The solid then fell from the tube to the bottom of the trap and melted, and the liquid was distilled into a cylinder cooled with liquid air, where it could be stored for future use. The pressure in the trap was observed on a manometer during distillation and kept near 1 atmosphere by partially closing the valve to the receiving cylinder. The last 5 or 10 percent of the liquid was discarded.

As judged by the narrowness of the melting range, this simple purifying procedure was adequate, and we may conclude that either the starting material was very pure or the impurities it contained were easily removed.

III. VAPOR PRESSURE

Vapor pressures below 2 atmospheres were measured with the N_2O in an adiabatic calorimeter.² The filling tube of the calorimeter transmitted the pressure to a mercury manometer which gave either the absolute pressure, or the excess above barometric pressure, as desired. Column heights were reduced to 0 °C in the usual way, and were reduced to standard gravity by multiplying by the factor 980.10/-980.665.

All vapor pressures above 2 atm, and a few which overlapped the manometer data, were measured with a 12-inch Bourdon gage. This gage, which had a range of 0 to 11 atm, was calibrated before and after the measurements. The latter calibration was the more thorough, and was made without resetting the zero of the pointer. It was therefore used in reducing the results. Comparison of the original and final calibrations did not show any lack of stability in the gage. All oil was washed from the gage before use.

The bulb that contained the liquid N_2O was a thin-walled monel tube $\frac{3}{16}$ by 20 inches, the lower part of which was immersed in the stirred liquid cryostat described by Scott and Brickwedde [2]. Connections to the gage and to the cylinder of purified N_2O were made with copper tubing (0.1 in. od). Temperatures were measured with platinum resistance thermometer *G*, previously calibrated [3] on the International Temperature Scale. This thermometer was placed in

² This calorimeter was first used by Scott, Meyers, Rands, Brickwedde, and Bekkedahl in the measurement of the vapor pressure and other thermodynamic properties of 1,3-butadiene.

the liquid bath beside the bulb containing the liquid N₂O. Data were taken by raising the temperature of the bath to the desired value and reading the gage at intervals until it was apparent that equilibrium had been reached. Usually there was no significant change during this period.

The results of the vapor pressure measurements are given in table 1. The Antoine equation

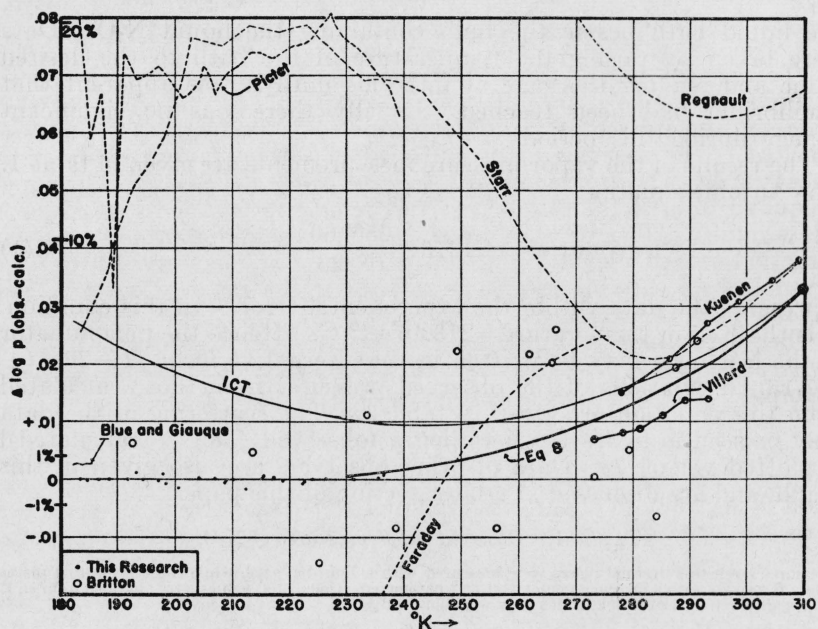
$$\log_{10} p(\text{atm}) = 4.1375 - \frac{656.60}{T - 26} \quad (1)$$

represents the data within the experimental error. In this equation, T is the Kelvin temperature $= 273.16 + t^{\circ} \text{C}$. Hence the denominator of the last term may be written in the alternative forms $T - 26 = t + 247.16$. Deviations of the observed pressures from those calculated from this equation are given in table 1. The scattering of the data may be seen in figure 1, where $[\log p(\text{observed}) - \log p(\text{calculated})]$ is plotted versus T . Data of other observers are also given in this graph, and are discussed in a later section of this paper.

TABLE 1.—Observed vapor pressures of N₂O

[Pressures given to 3 decimal places were measured with a Bourdon gage, while those given to 4 places were measured with a mercury manometer and barometer. Calculated values were obtained from eq 1. Data are given in the order in which they were obtained]

T	$p(\text{obs})$	$\Delta p(\text{obs.} - \text{calc.})$
$^{\circ} K$	atm	atm
198.41	2.130	−.004
204.14	2.831	.002
208.71	3.495	−.003
212.18	4.066	−.015
215.18	4.645	.003
218.92	5.417	−.003
222.59	6.269	−.006
226.44	7.275	.002
231.03	8.612	.002
236.86	10.589	.031
197.98	2.092	.005
208.88	3.524	−.001
215.31	4.674	.006
222.41	6.222	−.008
231.48	8.752	.000
236.54	10.464	.019
193.31	1.640	.007
200.61	2.375	−.008
206.95	3.229	.002
210.11	3.726	.000
217.12	5.038	.003
224.04	6.637	.000
230.63	8.499	.011
235.61	10.120	.004
183.313	0.9200	.0002
184.625	.9955	−.0004
186.578	1.1184	.0003
188.425	1.2447	.0002
191.703	1.4966	.0004
194.407	1.7324	.0002
197.048	1.9891	−.0007

FIGURE 1.—Vapor pressure of N_2O .

The ordinates are deviations of observed values from those calculated from the equation $\log_{10} p(\text{atm}) = 4.1375 - \frac{656.60}{T-26}$. This equation represents the data obtained in this research within the experimental error but at higher temperatures it gives pressures that are too low.

Table 2 contains values of p computed from equation 1 at 5-degree temperature intervals. It is hoped that the vapor pressures in this table are correct to 1 percent or better throughout the entire range. Where the mercury manometer was used (below 2 atm) they are of course much better.

TABLE 2.—Vapor pressures and latent heats of vaporization of liquid N_2O , at uniform temperature intervals.

[Pressures were computed from eq 1, latent heats from eq 5. Molecular weight=44.016.]

T	p	L	L
$^{\circ}K$	atm	$kJ\ mole^{-1}$	$J\ g^{-1}$
^a 182.351	0.8670	16.66	378.6
^b 184.695	1.0000	16.55	376.0
185	1.018	16.54	375.7
190	1.361	16.29	370.2
195	1.788	16.04	364.4
200	2.312	15.78	358.4
205	2.946	15.50	352.1
210	3.707	15.21	346
215	4.607	14.91	339
220	5.662	14.59	331
225	6.887	14.26	324
230	8.297	13.91	316
235	9.906	13.54	308
240	11.730	13.15	299

^a Triple point.

^b Boiling point.

IV. TRIPLE POINT AND BOILING POINT

To test the purity of the N_2O , and to determine the triple point temperature, a portion of the purified material was placed in the calorimeter and its temperature determined as a function of the fraction melted. The measurements included a determination of the gross heat capacity both above and below the triple point, so that the heat required to melt the sample without raising its temperature could be determined. The value thus obtained was used in computing the fraction melted, but did not lead to a value of the heat of fusion per gram, as no suitable means of measuring the mass of material was available at the time.

Temperature as a function of fraction melted is shown in figure 2, where the two types of circles refer to two different runs on the same sample. The equation of the curve in the figure is

$$182.351 - T = (RT_f^2/L_f)(7.10^{-6}/F), \quad (2)$$

where T_f is the temperature of fusion, L_f the latent heat of fusion, and F the fraction of the material melted. Assuming this equation to represent the depression of the melting point by impurity, the mole

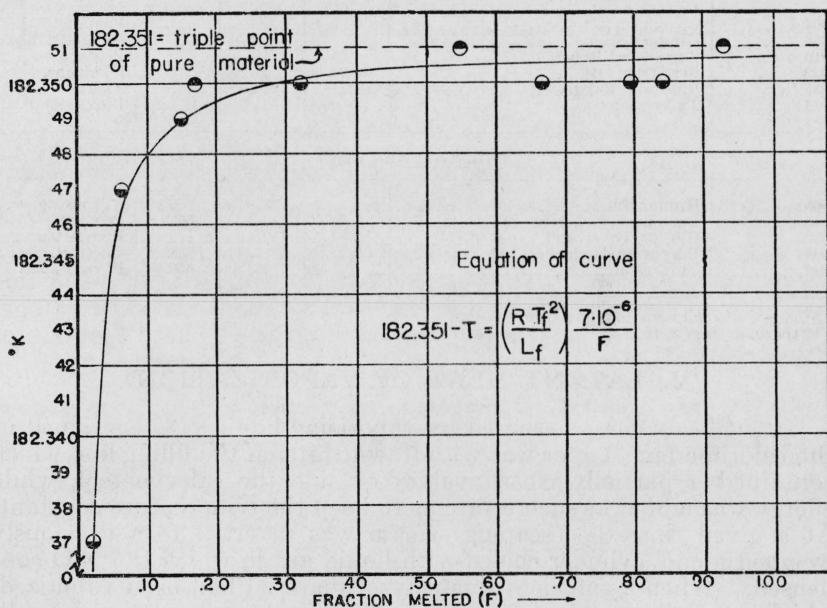


FIGURE 2.—Temperature of a sample of N_2O as a function of the fraction of the material melted.

fraction of impurity is 7×10^{-6} , and the melting point of pure N_2O is 182.351°K . Blue and Giauque's [1] value for the latent heat of fusion was used ($1563.0 \text{ cal mole}^{-1}$). The triple-point pressure, found by substituting 182.351°K in equation 1 is 0.8670 atm , or 658.9 mm Hg . The boiling point, calculated from the same equation, is 184.695°K . These values are probably correct within ± 5 millidegrees on the International Temperature Scale. The temperature measurements

were made with platinum resistance thermometer *B*, which was calibrated in the same way [3] as thermometer *G*, used in the measurement of vapor pressures above 2 atm.

The triple point and boiling point of N_2O give promise of being satisfactory thermometric fixed points at which the calibration of thermometers may be checked. In view of their possible usefulness for this purpose, values of the triple- and boiling-point temperatures reported since 1900 have been assembled in table 3. The triple-point pressure is included where available. A more exhaustive study would be required to establish the reliability of these two fixed points under all conditions likely to arise in practice, but there is as yet no indication that they would be unsatisfactory.

TABLE 3.—*Values of the triple point and boiling point of N_2O published since 1900*

[Temperatures are given as reported by the authors, without conversion from centigrade to absolute or vice versa]

Date	Reference	<i>t</i>	<i>T</i>	<i>p</i>	Thermometer
N_2O, BOILING POINT					
1904.....	Grunmach [21].....	°C -89.4	°K 183.9	mm Hg 741.1	Pentane.
1906.....	Hunter [22].....		186.0		^a Pt resistance.
1916.....	Burrell and Robertson [23].....	-88.7	184.4		Pentane.
1922.....	Bergstrom [24].....	-89.5			^b Toluene.
1935.....	Blue and Giauque [1].....		184.59		Thermal.
1944.....	This research.....		184.695		Pt resistance.
N_2O, TRIPLE POINT					
1906.....	Hunter [22].....		183.9	660	^a Pt resistance.
1916.....	Burrell and Robertson [23].....	-90.6	182.5	666	Pentane.
1930.....	Clusius, Hiller, and Vaughan [25].....		182.4		Pb resistance.
1935.....	Blue and Giauque [1].....		182.26	658.96	Thermal.
1944.....	This research.....		182.351	658.9	Pt resistance.

^aUsing Callendar equation, no β -term.

^b2 thermometers were used.

V. LATENT HEAT OF VAPORIZATION

Latent heats were measured by vaporizing liquid N_2O contained in the calorimeter. Vapor was withdrawn through the filling line, which contained a partially closed valve (outside the calorimeter), while energy was added at such a rate as to keep the temperature constant. At a given time the escaping stream was diverted to a previously weighed monel cylinder cooled with liquid air, in which the N_2O condensed. When a sufficient quantity of material had been vaporized, which generally required from 15 to 30 minutes, the stream was diverted to another receiver. The monel cylinder was then allowed to warm to room temperature and the increase in weight determined. Since the cylinder had to withstand the vapor pressure of N_2O at room temperature (about 55 atm), it was necessarily rather heavy (weight empty 956 g, internal volume 324 cm³). During the weighings an airtight counterpoise, which displaced approximately the same volume of air as the cylinder, was used to minimize errors due to changes in the buoyant force of the air.

The results are given in table 4. The molar latent heat is given by

$$L = 44.016 (Q/M) (1 - V_l/V_g), \quad (3)$$

where 44.016 is the molecular weight; and M is the mass of N₂O expelled from the calorimeter by addition of the energy Q . The quantities V_l and V_g are the molar volumes of the liquid and vapor phases under the conditions of the experiment, and the factor $(1 - V_l/V_g)$ takes account of the vaporized material that remains in the calorimeter to fill the space formerly occupied by liquid. The correction for vapor remaining in the calorimeter is relatively unimportant in the range covered by the present measurements, as may be seen from the values of $(1 - V_l/V_g)$ given in table 4. Values of V_l were taken from reference [4], whereas values of V_g were computed from an equation proposed by Meyers [5], which may be written in the form

$$(1 - V_g/V_l) (1 - V_l/V_l) = (p/ep_c)^{0.7}. \quad (4)$$

In this equation, V_g and V_l are, as above, the molar volumes of saturated vapor and liquid in equilibrium with each other, and V_l is RT/p , the volume which an ideal gas would occupy under the same conditions. Also, p is the vapor pressure and p_c the critical pressure of the substance, taken in this case to be 71.7 atm [6], and e is the base of natural logarithms.

TABLE 4.—Experimentally determined latent heats of vaporization

[M is mass of gas expelled from calorimeter by addition of energy Q . From these the molar latent heat L is obtained with the aid of eq 3]

T	$p(\text{calc.})$	M	Q/M	$(1 - V_l/V_g)$	L	$\Delta L(\text{obs.} - \text{calc.})$
$^{\circ}\text{K}$	atm	g	$j \text{ g}^{-1}$		kJ mole^{-1}	kJ mole^{-1}
185.458	1.047	2.508	377.1	0.9974	16.55	0.03
187.243	1.163	11.854	374.1	.9972	16.42	-.01
188.535	1.253	3.956	371.9	.9970	16.32	-.05
184.369	0.9806	16.949	377.8	.9976	16.59	.02
194.944	1.782	11.402	367.1	.9957	16.09	.05
200.445	2.364	9.449	359.9	.9944	15.75	.00
204.945	2.939	18.793	354.3	.9930	15.48	-.02
193.135	1.618	14.232	367.9	.9961	16.13	-.01
194.666	1.756	12.643	366.7	.9958	16.08	.02
196.419	1.926	12.550	363.8	.9954	15.94	-.03
198.643	2.159	13.289	361.3	.9948	15.82	-.03
185.910	1.075	12.242	375.6	.9974	16.49	.00

The experimental data are represented within the accuracy of the measurements by the equation

$$L(\text{kJ mol}^{-1}) = 1.571(T_c - T)^{0.57} - 0.0646(T_c - T), \quad (5)$$

where T_c is the critical temperature, taken to be 309.7°K [6].

Deviations of the observed values from this equation are given in table 4. The experimental points are plotted as circles in figure 3; the solid line corresponds to equation 5. This equation represents

both the experimental values given in table 4 and also values of L at higher temperatures, which were computed from vapor-pressure data with the aid of Clapeyron's equation.

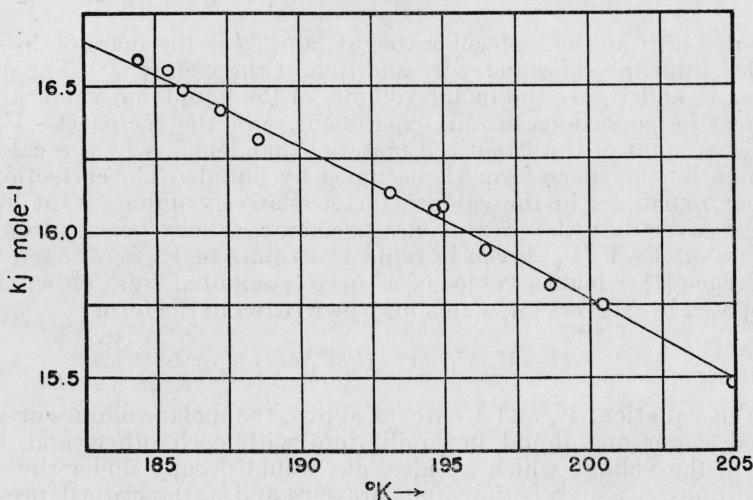


FIGURE 3.—Heat of vaporization of N_2O .

VI. COMPUTATIONS OF LATENT HEAT FROM CLAPEYRON'S EQUATION

Clapeyron's equation, written in the form

$$L = T \frac{dp}{dT} (V_g - V_l), \quad (6)$$

was used to compute values of L at 5-degree intervals throughout the entire range of the vapor-pressure measurements, which was approximately 183° to 240° K. Below 205° K the computed values could be compared with the directly observed values of L , and furnished a test of the consistency of the experimental measurements of vapor pressure and latent heat. Above 205° there were no direct observations of L , and equation 6 gave values not otherwise available. Values of dp/dT were obtained from equation 1. The values of V_l were taken from reference [4].

The most uncertain quantity entering into the calculation appeared to be V_g . Two methods were used for obtaining V_g , the first of which was based on work of Johnston and Weimer [7]. These authors used a gas thermometer to measure the second virial coefficient³ of N_2O . When vapor volumes computed from their data were used in equation 6, values of L were obtained that were about 4 parts per thousand lower than the observed values. Vapor volumes were also

³ The equation which they give for the second virial coefficient contains a misplaced decimal point. The coefficient of the term in $1/T$ must be multiplied by 10 in order to obtain agreement with the graph. This correction is given in J. Am. Chem. Soc. 57, 2737 (Dec. 1935).

obtained from equation 4. When these were used, somewhat better agreement was obtained, the values of L being within about 1 part in a thousand of the observed values.

An equation of the form

$$L = A(T_c - T)^\alpha + B(T_c - T) \quad (7)$$

was chosen to represent both the experimental values and those at higher temperatures calculated from equations 1, 4, and 6. Equation 7 gives a suitable temperature dependence of L near the critical temperature, and has been found to give a satisfactory representation of the latent heats of a number of substances. From equation 7 we may readily derive the equation

$$\log [L + (T_c - T)dL/dT] = \log[(1 - \alpha)A] + \alpha \log(T_c - T)$$

so that by plotting $\log [L + (T_c - T)dL/dT]$ versus $\log (T_c - T)$, a straight line of slope α should be obtained. Since the direct observations covered too small a range to determine α with any great accuracy, the values of L calculated from equation 6, with vapor volumes from equation 4, were used for this purpose. The derivative dL/dT was computed by the method of Rutledge [8], and the logarithmic plot gave the value 0.57 for α . The constant A could also have been obtained from this graph, but it was thought preferable to determine both A and B from the directly observed values of L . This led to equation 5, which was used to compute the values of L given in table 2. On comparing these with the values of L used in the determination of α , the greatest difference was found to be less than 1 part in a thousand. The agreement is considered to some extent fortuitous.

It is thought that the latent heats of vaporization given in table 2 are within 3 parts in a thousand of the true values in the range when direct determinations were made (up to 205°K). Above 205°K the uncertainty progressively increases, and may approach 2 percent at the highest temperatures.

VII. COMPARISON WITH PREVIOUS WORK

Cragoe [4] has analyzed the data on the vapor pressure of liquid N_2O extant at the time of publication of the International Critical Tables, and derived from them a table of values which is represented by the curve in figure 1 marked ICT. At the lower temperatures this table now appears to give pressures that are too high, primarily because the temperature assigned to the boiling point was too low. At the time of that analysis there were no satisfactory data at intermediate pressures. Since that time measurements have been made by Britton [9] and by Blue and Giauque [1]. Their results are shown in figure 1. Britton's values scatter considerably, but show no systematic deviation from those found in the present research.

Blue and Giauque measured vapor pressures of both solid and liquid, but covered a range of only 3.6 degrees in the liquid. The triple-point pressure reported by them and that found by the author are in excellent agreement, as may be seen from table 3. In view of

this, it may at first appear surprising that on figure 1 their pressures are more than 5 parts in a thousand higher than those of the author. The reason for this is almost certainly a difference in temperature scales. Blue and GIAUQUE give 182.26° K for the triple point. This value was obtained with a thermocouple which they estimate may be in error by several hundredths of a degree, although greater accuracy in the measurement of small temperature intervals was obtained by use of a gold resistance thermometer which was calibrated from time to time by comparison with the thermocouple. The thermocouple was originally calibrated by comparison with a hydrogen gas thermometer. Their triple-point temperature is 0.09 degree lower than that found in the present research, and raising all of their temperatures by this amount removes the small discrepancy between their results and the author's.

Although agreement of temperature scales within 0.09 degree may be considered satisfactory in many applications, it is of interest to consider the possible causes of the discrepancy in the present instance. Part of the discrepancy can be accounted for by the fact that the author's temperatures are based on $T_0=273.16^{\circ}$ K, whereas Blue and GIAUQUE used $T_0=273.1$. When this difference is taken into account the discrepancy is reduced by a maximum of 0.06 degree. The remaining 0.03 degree must be accounted for by errors in the two scales or by a difference between the International Temperature Scales and the thermodynamic temperature scale. In this temperature region the International Scale is believed to be reproducible to better than 0.01 degree. The remaining discrepancy could conceivably be due to a difference between the International Scale and the thermodynamic, but it is opposite in sign to the values of this difference previously reported [10, 11].

The range covered by figure 1 has been extended from 240° K to the critical point primarily for the benefit of those who may require values of the vapor pressure beyond the range of the present work. The most reliable data in this range appear to be those of VILLARD [12] and KUENEN [13]. The fact that these data give positive deviations in figure 1 is to be expected, as the Antoine equation is known to give pressures that are too low in the critical region when the constants are determined from data at lower temperatures.

Data from STARR [14], PICTET [15], REGNAULT [16] and FARADAY [17] are also shown in figure 1. None of these sets of data shows the correct temperature dependence, and little or no weight can be given to them. Although STARR's table is in error by almost 20 percent over part of the range, it has found its way into tables of refrigerating data [18], and as recently as 1941 appeared in the Chemical Engineers' Handbook [19]. It is interesting to note that the latter reference also contains (p. 375) the ICT vapor-pressure table, which gives more nearly correct values. It is not clear whether STARR made measurements himself or whether he based his table on earlier work.

The heavy curve in figure 1 extending from 230° K to the critical point was drawn to represent the probable course of the vapor-pressure curve in this region.

This curve represents the vapor-pressure equation

$$\log p = 4.52620 - (1/T)[829.148 + 1.157\gamma 10^{-4}(10^{3.4\gamma/2} 10^{-10} - 1)] \quad (8)$$

where $\gamma = 69,500 - T^2$, and $T = 273.16 + t^\circ \text{C}$. The author is indebted to C. H. Meyers for deriving this equation. He has used this form of equation with success for representing the vapor pressures of CO_2 [20] and other substances for which accurate data were available. Equation 8 may be used alternatively with equation 1 in the range covered by the author's data, as the two equations agree within less than the experimental error. Equation 8 would appear to give as reliable values of vapor pressure above 240°K as present data permit, but subsequent measurements may well show these to be substantially in error at the higher pressures.

The critical temperature and pressure given by equation 8 are those accepted by Pickering [6]: $p_c = 71.7 \text{ atm}$, $t_c = 36.5^\circ \text{C}$, which agree with those given in reference [4].

For the heat of vaporization at 1 atmosphere, Blue and Giauque [1] give $3958 \pm 3 \text{ cal mole}^{-1}$. Converting this to international joules, we find $16.557 \pm .013 \text{ kJ mole}^{-1}$, which is to be compared with $16.55 \text{ kJ mole}^{-1}$ found in the present investigation.

VIII. TABLES IN ENGINEERING UNITS

For the convenience of those who prefer to use engineering units, data given in the preceding sections of this paper have been converted with the aid of the following data:

$$f(^{\circ}\text{F}) - 32 = 1.8[T(^{\circ}\text{K}) - 273.16].$$

$$1 \text{ atm} = 14.696 \text{ psi.}$$

$$1 \text{ J g}^{-1} = (1.8 \times 860)/3600 \text{ Btu lb}^{-1} \\ = 0.43000 \text{ Btu lb}^{-1}.$$

By substituting these relations into equations 1 and 5, we obtain the alternative forms

$$\log_{10} p (\text{psi abs}) = 5.3047 - \frac{1181.88}{f + 412.89} \quad (1a)$$

and

$$L (\text{Btu lb}^{-1}) = 10.978(f_c - f)^{0.57} - 0.3506(f_c - f), \quad (5a)$$

where f is the temperature in degrees fahrenheit, and f_c , the critical temperature, is 97.8°F . Equations 1a and 5a have been used to compute values of p and L at intervals of 10 degrees fahrenheit between the triple point and -30°F (238.716°K). These are given in table 5. Above -30°F , vapor pressures have been computed with the aid of equation 8. It is emphasized again that the present investigation did not extend above -30°F and that vapor pressures in this region are based on an analysis of previously published work.

TABLE 5.—Vapor pressures and heats of vaporization of liquid N₂O in engineering units

Temperature	Pressure	Heat of vaporization
° F	psi(abs)	Btu lb ⁻¹
a—131.456	12.741	162.8
b—127.237	14.696	161.7
—130	13.39	162.4
—120	18.60	159.8
—110	25.28	157.1
—100	33.68	154.2
—90	44.10	151.2
—80	56.8	148.0
—70	72.1	144.7
—60	90.3	141.2
—50	111.6	137.5
—40	136.5	133.6
—30	165.2	129.5
—20	199.0	
—10	236.8	
0	279.7	
10	328.0	
20	382.1	
30	442.4	
40	509	
50	583	
60	664	
70	754	
80	853	
90	962	
c 97.8	1,054	

a Triple point.

b Boiling point.

c Critical point.

IX. REFERENCES

References [1] to [25] are referred to in the text and tables of this paper. The remainder were located when a somewhat more extensive study of N₂O was in prospect, and are included here because of their close relation to the present work.

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